Powder Size Standards

This research is directed toward a development of standard reference materials for powder size measurement. Research is extending measurement capabilities to sizes to less than 1 micrometer and a broader range of materials for different applications.

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We develop and certify glass/ceramic powders as particle size distribution, PSD, Standard Reference Materials (SRMs). These SRMs are needed in industry for instrument calibration, quality control in powder manufacture, and for compliance with ISO 9000 requirements. Necessary to this certification is the development of sampling protocols and size measurement procedures. The primary standards for particle size distribution are a series of glass bead SRMs covering the range from micrometer to millimeter. Maintaining and improving these references is an ongoing effort. Requirements have been identified for several industry specific particle size distribution standards. This year, two reference materials were completed for the thermal spray industry (SRMs 1984 and 1985). These represent the two major WC powder types used by that industry: a fused and crushed powder, and a spray dried and sintered material.

The SRM 1004b (Glass beads with particle size range of 38 µm to 125 µm) has been certified for size distribution this year by scanning electron microscopy/computer assisted image analysis. Figure 1 illustrates the PSD for SRM 1004b .

The impact of this work is the availability to industry of a variety of materials and size ranges for particle size standards. There are approximately five hundred (500) units sold each year of the particle size SRMs. Research is underway for the development of glass bead SRMs covering the size range from 1 µm to 50 µm.

A cooperative effort with CENAM has been established with a visiting scientist, Mario Cordero to develop a particle size distribution certified reference material for Mexico.

There is growing interest in the use and application of submicron and nanosized powders. However, the use of such powders is significantly limited by the ability to reliably characterize the particle size distribution of these systems. Efforts have focussed on exploring new techniques and instruments for determination of size distribution. A technique of particular interest that has been studied is based on the settling of particles under the action of centrifugal forces. Various sub-micrometer ceramic particulate systems including silica, alumina, titania, and ceria have been examined by this technique. Influence of techniques of specimen preparation on the observed results have been studied and interpreted. Instrument sensitivity to the state of specimen dispersion, concentration and particle size has been examined. Experience gained through these tests will be utilized in efforts for the development of particle size SRMs in the sub-micrometer range.

A descriptive text covering important issues encountered during particle size determination has been published as a NIST Recommended Practice Guide. This guide is a compilation of essential facts and some fundamental information about commonly used techniques of particle size analysis in the ceramics industry.

Industrial users have expressed the opinion that such documents give them easy access to varied information about particle size determination in one source. References to national and international standards were of particular interest to all the reviewers. A major raw material producer has expressed interest in providing its national and international customers this practice guide to use as a common basis for comparison of size results in cases of significant discrepancies between the producer and the customer.

NIST testing of SRM 1897 has been completed and a round robin testing program for final certification has begun. Completion of this SRM will conclude a series of new materials available from NIST at the level of SRM certification. The availability of SRM level materials will enable users to more easily certify their instrumental calibrations with increasingly strict national and international standards for performance and traceability of results.

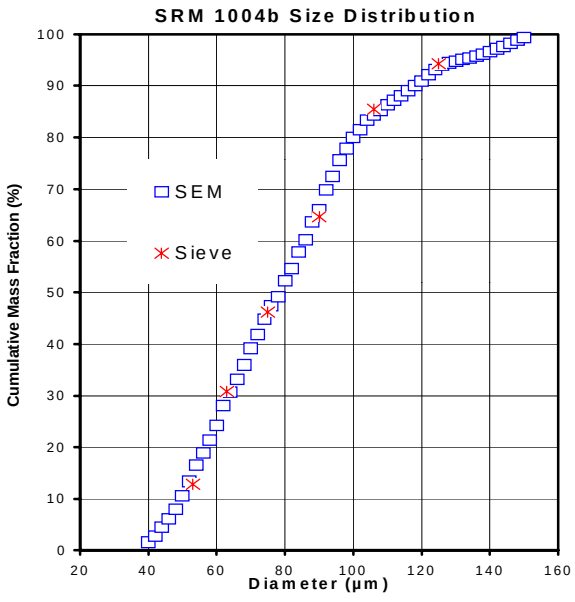

Figure 1. SRM 1004b size distribution

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